भारतीय मानक

Indian Standard

IS 4394 : 2023

वस्त्र रंजक सामग्री — सजातीय वैट रंजक सामग्री की ताकत के निर्धारण की विधि

(दूसरा पुनरीक्षण)

Textile Dyestuffs — Method for Determination of Strength of Homogeneous Vat Dyestuffs

(Second Revision)

ICS 59.040; 71.040.50

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December 2023 **Price Group 6**

Textile Speciality Chemicals and Dyestuffs Sectional Committee, TXD 07

FOREWORD

This Indian Standard (Second Revision) was adopted by the Bureau of Indian Standards, after the draft finalized by the Textile Speciality Chemicals and Dyestuffs Sectional Committee had been approved by the Textiles Division Council.

Vat dyes are marketed in a large number of varieties in powder, liquid or paste form with different strengths. They are mainly used for colouring cellulosic fibres and most favoured for applications wherever the fastness demands are of prime importance.

The evaluation of the strength of a dye is useful for the manufacturer to standardize the product batches and for the user for matching of shades, evaluating cost-benefit ratio and adjustment of dyeing recipes to minimize dyeing irregularities.

The method prescribed in this standard is a general method and wherever special instructions are given by the manufacturers, these should be followed while carrying out the dyeings. In order to compare the strength of the dyestuff under test and the standard dyestuff, dyeings of standard depths are recommended as given in Table 1.

The present revision of the standard has been made in the light of experience gained since its publication and to incorporate the following major changes:

a) Title of the standard has been modified;
b) Grade and purity of chemicals used have been specified;
c) Sampling clause has been modified;
d) D65 light source has been specified in place of north skylight for evaluation of dyed material;
e) Vat yellow paper has been specified in place of vat yellow G paper;
f) For dyeing method A/Q^{1+}, the provision for manufacturer recommendations have been provided for vatting and dyeing conditions;
g) Hydrogen peroxide, acetic acid, and sodium hypochlorite have been additionally specified as dyeing assistant to align the dyeing procedure with current practices, accordingly dyeing procedure has been modified; and
h) References to Indian Standard have been updated.

The composition of the Committee responsible for the formulation of this standard is given in Annex C.

In reporting the results of a test or analysis made in accordance with this standard, if the final value, observed or calculated, is to be rounded off, it shall be done in accordance with IS 2 : 2022 ‘Rules for rounding off numerical values (*second revision*)’.

Indian Standard

TEXTILE DYESTUFFS — METHOD FOR DETERMINATION OF STRENGTH OF HOMOGENEOUS VAT DYESTUFFS

(Second Revision)

1 SCOPE

1.1 This standard prescribes a method for evaluating strength of the homogeneous vat dyestuffs by exhaust dyeing.

1.2 The method prescribed in this standard is not applicable to mixtures of dyestuffs. The qualitative tests are given in Annex A to distinguish a homogenous dyestuffs (with or without corrective shade components) from a mixture of dyestuff.

2 REFERENCES

The standards given below contain provisions which through reference in this text, constitute provision of this standard. At the time of publication, the editions indicated were valid. All standards are subject to revision and parties to agreements based on this standard are encouraged to investigate the possibility of applying the most recent edition of these standards:

IS No.	*Title*
IS 1070 : 2023	Reagent grade water — Specification (*fourth revision*)

3 TERMINOLOGY

For the purpose of this standard, the following definitions shall apply:

3.1 Depth of Shade — The amount of dyestuff in gram in the dye bath per 100 g of the fibre, expressed as percentage.

3.2 Strength of Dyestuff — The percentage ratio of the strength of dyestuff under test to that of the standard dyestuff, the strength of which is assumed to be 100 percent.

3.3 Liquor Ratio — The ratio of the mass of the material to be dyed to the mass of the liquor (dye solution or dye bath in which the material is to be dyed).

4 STANDARD DYESTUFF

4.1 The standard sample of dyestuff, against which the strength of dyestuff under test is evaluated, shall be as agreed to between the buyer and the seller.

5 QUALITY OF REAGENTS

Unless otherwise specified analytical reagent grade chemicals with 99.0 percent purity shall be employed in tests and distilled water (*see* IS 1070) shall be used where the use of water as reagent is intended.

6 DETERMINATION OF STRENGTH OF DYESTUFF

6.1 Carry out the qualitative tests given in Annex A to determine whether the dyestuff under test is a mixture of dyestuffs or a single homogeneous dyestuff (with or without corrective shade components added).

NOTE — Two qualitative tests are prescribed in Annex A. If necessary, both the tests should be carried out to find out the components in the mixture of dyestuffs. If corrective shade components are added to the dyestuff, they are also detected by these tests.

6.2 Out of the sets of conditions of test laid down in Table 1, choose the set of conditions applicable to the dyestuff under tests.

6.3 Prepare dyeings for both the recommended percentages of standard sample of dyestuff (*see* **4.1**) by following the procedure given in Annex B; prepare simultaneously additional dyeings of the standard sample, with the percentage variations of dyeing strength by 5 percent on either side of each recommended percentage.

6.4 Simultaneously, prepare dyeings of different percentages of the dyestuff under test by following the procedure given in Annex B.

6.5 Compare the dyeings obtained as in **6.4** with the dyeings obtained in **6.3**. Select a dyeing of the dyestuff under test which visually appears to be equal in strength to the dyeing of the standard dyestuff for each recommended percentage and note the percentage of these dyeings.

NOTES

1 The dyeings should be compared under D65 light source. The evaluation of the hanks dyed with the standard dyestuff and the dyestuff under test should be carried out by at least three observers for consistency in the results. This may also be done instrumentally, if facilities exist.

2 Before comparing the dyeings, they should be spread out properly. The consistency in different dyeings of standard dyestuff and the sample of dyestuff under test should be observed. If the strength variations between the two consecutive dyeings are not constant, the dyeings should be repeated.

Table 1 Vatting and Dyeing Conditions for Test

(*Clauses* 6.2, B-5.1 *and Note under* B-5.3.1)

Sl No.	Dyeing Method	Dyeing Percentage	Vatting Conditions					Bulking and Dyeing Conditions				
			Quantity of Dyestuff (g)	Vatting Volume (ml)	Sodium Hydroxide 53° Tw (ml)	Sodium Hydro Sulphite (g)	Vatting Temperature (°C)	Sodium Hydroxide 53° Tw (ml)	Sodium Hydro Sulphite (g)	Sodium Chloride (g)	Total Volume of Dye Liquor (ml)	Dyeing Temperature (°C)
(1)	(2)	(3)	(4)	(5)	(6)	(7)	(8)	(9)	(10)	(11)	(12)	(13)
i)	A/Q[1]	1.0 and 2.5	0.1 or 0.25	75	4.5	1.5	55 to 60	1.5	0.5	–	300	55 to 60
ii)	A/Q[2]	1.0 and 2.5	0.1 or 0.25	75	2.0	1.5	45 to 50	1.5	0.5	6.0	300	45 to 50
iii)	A/Q[3]	1.0 and 2.5	0.1 or 0.25	75	2.0	1.5	35 to 40	1.5	0.5	10.0	300	25 to 30
iv)	A/Q[1+]	1.0 and 2.5	0.1 or 0.25	75	*	*	55 to 60	*	*	*	300	60 to 65

NOTES

1 The temperature mentioned in col (8) and col (13) refer to the temperature of dye liquor in the dye vessels and not to the temperatures of water bath.

2 The conditions given in Table 1 are for guidance only. Wherever special instructions are supplied by the Manufacturer, these should be followed.

3 For a particular dye, the method giving maximum yield, as recommended by the manufacturer, should be followed.

4 The recommended percentage dyeings of standard dyestuff is shown under col (3). Suitable dyeing percentage of the dyestuff under test should be employed to obtain a shade within the range of standard dyeings, which is convenient for visual comparison.

5 The vatting, bulking and dyeing conditions would remain unchanged within 10 percent in the percentages of dyeings taken.

*Manufacturer recommendation shall be followed.

6.6 Calculate the strength of the dyestuff under test for each recommended percentage by the following formula:

$$S = \frac{A}{B} \times 100$$

where

S = strength of dyestuff under test, in percent;

A = percentage dyeing of standard dyestuff; and

B = percentage dyeing of dyestuff under test (*see* **6.5**).

6.7 Calculate the average of the two values obtained in **6.6**.

7 REPORT

7.1 Report the value obtained in **6.7** as the strength of the dyestuff under test as compared to the standard dyestuff.

8 SAMPLING

8.1 Lot

All the containers of the same dyestuff and of the same strength delivered to a buyer against one dispatch note shall constitute a lot.

8.2 Unless otherwise agreed to between the buyer and the seller, the number of containers to be selected at random from a lot shall be as given in Table 1.

Table 1 Sample Size

(*Clause* 8.2)

Sl No.	Lot Size	Sample Size
(1)	(2)	(3)
i)	2 to 15	2
ii)	16 to 25	3
iii)	26 to 50	4
iv)	51 to 100	5
v)	101 to 150	6
vi)	151 to 300	7
vii)	301 and above	8

8.3 If the dye is marketed in powder form, draw from each container selected in **8.2** a small quantity of the dye by a suitable sampling instrument from three different parts and mix thoroughly to get a composite sample weighing about 20 g. This shall constitute the test sample.

8.4 For dyes marketed in liquid emulsion or paste form, shake each container selected in **8.3** thoroughly so as to homogenize the dye dispersion and then draw the test sample as given in **8.3**.

ANNEX A

(*Clauses* 1.2 *and* 6.1)

QUALITATIVE METHOD FOR DETERMINATION OF COMPONENTS IN VAT DYESTUFF

A-1 PAPER CHROMATOGRAPHY TEST

A-1.1 Apparatus

A-1.1.1 *Two Plate Glasses* — evenly smooth, each of size 25 cm × 25 cm. The thickness of each of the plates shall be 1 cm. One plate shall have a centre hole of 1.5 cm diameter.

A-1.1.2 *Conical Flasks* — of 250 ml capacity

A-1.1.3 *Pipettes* — of 1 ml, 10 ml and 25 ml capacities

A-1.1.4 Watch glasses

A-1.1.5 *Measuring Cylinder* — of 100 ml capacity

A-1.1.6 Water bath

A-1.1.7 *Filter Paper* — of 3 mm thickness and of the size of plate glasses

NOTE — Whatman filter paper No. 1 (for chromatography) is suitable for this test.

A-1.2 Reagents

A-1.2.1 *Sodium Hydroxide Solution* — specific gravity 1.27 (or 53° Tw)

A-1.2.2 *Sodium Hydrosulphite* — of minimum 85 percent strength

A-1.2.3 *Pyridine* — boiling range 114 °C to 116 °C

A-1.2.4 Lint dust or any other suitable filtering aid

A-1.3 Procedure

A-1.3.1 Place the filter paper in between the two plate glasses, the one with centre hole coming on top. Pack tightly and uniformly half the height of the centre hole with lint dust.

A-1.3.2 Weigh about 0.05 g of the dyestuff under test on a watch glass. Transfer the dyestuff to 250 ml conical flask using 10 ml of distilled water. Add 2 ml of sodium hydroxide solution followed by 4 ml of pyridine. Make up the volume to 100 ml. Heat the contents in the flask in the waterbath to the vatting temperature given in Table 1. Add 0.5 g of solution hydrosulphite and vat the dyestuff for 10 minutes.

A-1.3.3 Prepare an eluting solution by taking 80 ml of distilled water in a 250 ml conical flask, and adding to it 5 ml of sodium hydroxide solution, 2.5 g of sodium hydrosulphite and 15 ml of pyridine in the same order. Stopper the flask with a rubber bung.

A-1.3.4 Put 1 ml dye solution (*see* **A-1.3.2**) on lint dust in the centre hole of the plate glass.

Immediately put eluting solution in the centre hole so that there shall be no oxidation of leuco vat dyestuff. Cover the centre hole with a small watch glass to avoid oxidation of leuco vat dyestuff by exposure to air. Continue the elution till the wet halo nearly reaches the limits of the filter paper (*see* Note). Remove the filter paper from the plate glasses. Dry the filter paper and simultaneously oxidize the leuco vat dye on it by hanging the filter paper from a peg in air. Examine the filter paper for the presence of components.

NOTE — During elution, the central portion of the filter paper should not be allowed to dry. The watch glass should be removed only for the addition of eluting solution. By the time the wet halo reaches the limits of the paper, the components to the mixture get clearly separated into different coloured bands. Even though the chromatograph obtained during elution would indicate the presence of components, the results should be assessed only on the oxidized dyestuff on chromatograph.

A-2 BLOW TEST

A-2.1 Apparatus

A-2.1.1 *White Enamelled Tray* — 25 cm × 20 cm × 4.5 cm in size

A-2.1.2 *Filter Paper* — of 3 mm thickness and 30 cm × 18 cm size

NOTE — Whatman filter paper No. 1 is suitable for the test.

A-2.1.3 *Glass Rod* — of 5 mm diameter

A-2.1.4 *Folded Filter Paper* — of size 10 cm × 1.5 cm and folded lengthwise

A-2.2 Reagents

A-2.2.1 *Sodium Hydroxide Solution* — sp gr 1.27 (or 53° Tw)

A-2.2.2 *Sodium Hydrosulphite* — of minimum 85 percent strength

A-2.2.3 Turkey red oil

A-2.3 Procedure

A-2.3.1 Pour 1 500 ml of distilled water at 70 °C to 75 °C into the clean enamelled tray. Add 30 ml of sodium hydroxide solution, 7.5 g of sodium hydrosulphite and 3 drops of Turkey red oil. Stir the solution well.

A-2.3.2 Introduce one filter paper (*see* **A-2.1.2**) into the tray. Take enough of dyestuff under test on the folded filter paper and blow it on the surface of the solution in the tray (*see* Note 1). Vat the dyestuff for 5 minutes to 10 minutes (*see* Note 2). Take out the filter paper from the tray. Wash it in cold running water. Oxidize the leuco vat dyestuff in air and dry the filter paper in an atmosphere free from dust. Examine the filter paper for the presence of colour components.

NOTES

1 While blowing the dyestuff, care should be taken so that the dyestuff is not allowed to fall as a lump on the surface of the solution in which case the identification of the constituents is rendered difficult.

2 Careful observation of leuco vat dyestuff would be useful in the identification.

ANNEX B

(*Clauses* 6.3 *and* 6.4)

GENERAL METHOD FOR DYEING VAT DYESTUFFS

B-1 APPARATUS

B-1.1 Dye Vessels — Porcelain or stainless steel beakers or dye vessels provided for mechanically agitated dye baths (*see* **B-4.3**).

B-1.2 Watch Glass

B-1.3 Graduated Pipette — capable of measuring correct to 0.1 ml.

B-1.4 Water Bath

B-2 DYEING ASSISTANTS

B-2.1 Water — Distilled water (*see* IS 1070) shall be used in the preparation of the dye bath (*see* **B-4.3**).

NOTE — For rinsing and soaping water having hardness of not more than 50 ppm expressed as calcium carbonate may be used.

B-2.2 Sodium Hydroxide Solution — sp gr 1.27 (or 53° Tw)

B-2.3 Sodium Hydrosulphite — of minimum 85 percent strength

B-2.4 Vat Yellow Paper

B-2.5 Phenolphthalein Paper

B-2.6 Wetting Agent — methylated spirit or highly sulphonated castor oil

B-2.7 Dilute Hydrochloric Acid — containing 2 ml/l of hydrochloric acid (sp gr 1.16)

B-2.8 Soap Solution — 5 g/l

B-3 PREPARATION OF HANKS FOR DYEING

B-3.1 Test Hanks

A sufficient number of hanks of scoured, bleached, unmercerized cotton yarn having no finishing chemical or blueing agent shall be used for the test. Each hank should weigh 10 g ± 0.1 g.

NOTES

1 Any yarn normally used in laboratories for carrying out dyeing trials or yarn of the following requirements is suitable for this test:

a) Count - 10 tex × 2 (or 60s/2);

b) Twist per metre — 750; and

c) Cuprammonium fluidity not more than 5 rhes.

2 If the mass of a hank is not 10 g ± 0.1 g, it should be weighed accurately and the amount of dyestuffs and chemicals to be taken should be calculated accordingly.

B-3.2 Preparation of Test Hanks

The hanks shall be treated in boiling water for 10 minutes, squeezed evenly to contain approximately its own mass of water, cooled and entered in the dye bath.

B-4 PROCEDURE

B-4.1 Vatting and Dyeing Conditions

The vatting and dyeing conditions for the recommended percentage dyeing shall be as given in Table 1.

B-4.2 Liquor Ratio

The liquor ratio shall be 1 : 30 in all operations; unless otherwise stated.

B-4.3 Preparation of the Dye Bath

B-4.3.1 Weigh accurately the requisite quantity of dyestuff in a watch glass and transfer it to the dye vessel with little water. Paste the dyestuff with necessary amount of wetting agent and add sufficient amount of hot water with constant stirring. Add more hot water to make up the volume to 75 ml for vatting. Add the recommended quantity of sodium hydroxide solution with constant stirring and heat the dye liquor to the recommended temperature for vatting. Add the recommended quantity of sodium hydrosulphite with constant stirring. Allow the dye to reduce at this temperature for 15 minutes to 20 minutes with occasional stirring.

NOTE — The required amount of dyestuff should be weighed accurately for each hank separately; in case a sensitive, balance for the purpose is not available, a large amount of dyestuff (such as 0.5 g) may be weighed and a stock vat of the dyestuff prepared. The quantities of sodium hydroxide and sodium hydrosulphite shall be such as so ensure complete reduction of the stock vat. Aliquot amounts of the stock vat shall be pipetted out for each dyeing as required. The final dyeing volume shall contain the recommended quantities of sodium hydroxide and hydrosulphite as in Table 1.

B-4.3.2 Prepare the bulk solution by adding the recommended quantities of sodium hydroxide

solution and sodium hydrosulphite in the remaining amount of water kept at the recommended temperature and add this to the dye bath with stirring.

B-4.4 Dyeing

Stir the dye liquor and enter the wetted hank (*see* **B-3.2**). Agitate the liquor to obtain a level dyeing. Dye the hank for 45 minutes at the recommended temperature agitating the dye liquor at frequent intervals. Care should be taken to keep the hank immersed below the surface of the liquor. Avoid excessive aeration of the hank to prevent oxidation of the reduced dye. Remove the hank from the dye bath. Squeeze it evenly and rinse in running cold water for 5 minutes. Squeeze the hanks and air oxidise for 10 minutes or, enter into a bath at 50 ºC containing 2 ml/l hydrogen peroxide (50 percent) and 0.5 ml/l to 1.0 ml/l acetic acid (80 percent) and treat for 10 minutes to 15 minutes. Remove the hank and wash it well in cold running water and soap it as in **B-4.5**.

NOTES

1 During dyeing, the liquor shall be tested frequently for the presence of sodium hydrosulphite and sodium hydroxide by spotting vat yellow paper and phenolphthalein paper respectively. Vat yellow paper turns blue in presence of sufficient amount of sodium hydrosulphite and phenolphthalein paper turns pink in presence of sodium hydroxide,

2 All dyeings should be carried out simultaneously in the same dye bath in separate vessels to ensure identical conditions.

3 For C.I. vat green 9, after cold rinse and air oxidation, the dyed hanks shall be developed for 15 minutes in a cold (20 ºC) aqueous solution of sodium hypochlorite containing 3 g/l available chlorine. The hanks are then washed thoroughly, soured with 5 ml/l hydrochloric acid (32º Tw) for 3 minutes, washed and soaped as usual.

B-4.5 Soaping

Enter the hank in a boiling soap solution (2 g/l) (*see* **B-2.8**) containing 2 g/l sodium carbonate, anhydrous and continue the treatment at boil for 15 minutes to 20 minutes with frequent stirring. Remove the hank and rinse it well in running water. Dry the hank in oven at a temperature not exceeding 70 °C.

ANNEX C

(*Foreword*)

COMMITTEE COMPOSITION

Textile Speciality Chemicals and Dyestuffs Sectional Committee, TXD 07

Organization	*Representative(s)*
Department for Jute and Fibre Technology Institute of Jute Technology University of Calcutta, Kolkata	PROF A. K. SAMANTA (***Chairperson***)
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Review of Indian Standards

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This Indian Standard has been developed from Doc No.: TXD 07 (21065).

Amendments Issued Since Publication

Amend No.	Date of Issue	Text Affected

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Published by BIS, New Delhi